

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

Application No. : 10/667,878  
Applicant(s) : Henry Drummond Boswell et al.  
Filed : September 22, 2003  
Title : Oxidative Treatment of Hair with Reduced Hair  
Damage  
TC/A.U. : 1615  
Examiner : Eisa B. Elhilo  
Conf. No. : 1156  
Docket No. : CM2517M2C  
Customer No. : 27752

**DECLARATION UNDER 37 CFR §1.132**

Commissioner for Patents  
P. O. Box 1450  
Alexandria, VA 22313-1450

Dear Sir:

I, Jennifer Mary Marsh, do hereby declare that I received a B.A. in Chemistry from Oxford University in Oxford, United Kingdom, and a Ph.D. in Inorganic Chemistry from Oxford University in Oxford, United Kingdom. I have been employed by The Procter & Gamble Company as a Principal Scientist from 1995 until present. I further declare that I have been involved in the development of hair colorant compositions for about seven (7) years.

I am a co-inventor of the above captioned patent application and am familiar with the subject matter contained therein. I am familiar with the Office Actions dated July 14, 2004, and December 14, 2004, and the references cited therein. I personally performed the experiments described below which contrast the lightening and the hair damage resulting

from treatment with compositions of the present application and with oxidative hair color compositions described in U.S. Patent No. 6,004,355 to Dias et al. ("Dias"). The emulsion base used in the experiments described below was made by Colin Clarke, who also is employed by the Procter & Gamble Company.

### EXPERIMENTAL METHODS

The following experiments were carried out to contrast the hair damage, as measured by the FT-IR Damage Assessing Protocol after a 5-Cycle Oxidative Hair Treatment Protocol With 2 Intermediate Washes, resulting in hair treated with compositions of the present application and with oxidative hair color compositions described in Dias.

The lightening and damage results of five products were contrasted for the present experiments. Products 1, 2, and 3 were made for the present experiments using a standard emulsion base as the carrier for the bleaching actives, as outlined in the Appendix 1. This emulsion base was the same as that disclosed in the present application. Products 4 and 5 were prepared, and lightening and damage data was generated for them, in association with previously conducted research relating to the present application. The ingredients of Products 4 and 5 also are outlined in Appendix 1. The five products can be described generally as follows:

Product 1: 0.15M peracetic acid + 1.18M hydrogen peroxide + 1.9% EDDS at pH 8

Product 2: 0.15M peracetic acid + 1.18M hydrogen peroxide + 0.1% EDTA at pH 8

Product 3: 0.15M peracetic acid + 1.18M hydrogen peroxide + 0.1% EDTA at pH 10

Product 4: 1.32M hydrogen peroxide + 2.0% EDTA at pH 10

Product 5: 1.32M hydrogen peroxide + 1.9% EDDS at pH 10

Reasons for choice of product formulations:

- All the products contained a total of 1.32M oxidant. This is the total level of oxidant used in the 5-Cycle Oxidative Hair Treatment Protocol With 2 Intermediate Washes as described in the present application.
- Products 1 and 2 were based on the Dias examples where a combination of hydrogen peroxide and peroxyacid precursors are used (in an 8:1 ratio). In these experiments,

we used peracetic acid, which is a common peroxyacid formed from the precursors, to ensure complete conversion in the emulsion base. The substantially same performance is expected from use of the pre-formed peroxyacid as that which would be expected if the precursors were used.

- Products 1 and 2 were made at pH 8 as in Dias, wherein the lower pH product is preferred in terms of lightening and damage.
- Product 3 was made with the same levels of hydrogen peroxide and peracetic acid as the oxidant as Products 1 and 2, but at the higher pH of pH 10.
- Products 2 and 3 were made with 0.1% EDTA, which is the level of chelant used in the Dias examples.
- Product 1 was made with 1.9% EDDS, which is the same as an exemplified level of chelant that may be used in accordance with the present application, and which is the level and chelant used in Product 5.
- No dyes were used in the products as we were measuring the lightening performance of the products.
- A standard level of alkaliser was used in all products. The alkaliser used was ammonia, which is not expressly mentioned in the Dias examples. However, experience has shown us that ammonia is a key component of the system to get efficient lightening and to achieve the desired pH. Further, the specification of Dias describes ammonium hydroxide as the preferred alkaline buffering agent.

For the treatment of the hair, 1.5g 'virgin' switches were used, sourced from Hugo Royer International Ltd, 10 Lakeside Business Park, Swan Park, Sandhurst, Berkshire, GU47 9ND, United Kingdom. "Virgin hair" means hair that has never been treated chemically. For each product, 6 of these 1.5g switches were used.

2g of the product per 1g of hair to be treated was applied on the hair switches and massaged in thoroughly. The hair switches were then wrapped in a plastic film and put in an oven at 30°C. After 30 minutes, the hair switches were removed from the oven and from the wrapping film, and were rinsed for 1 minute in water. 0.1g of shampoo per 1g of hair was then added and milked for 30 seconds at a rate of at least 150 strokes a minute before rinsing

for 30 seconds. The combined concentration of calcium and magnesium ions (water hardness) of the water used during all experiments (except for the preparation of the compositions tested, wherein water was distilled or dionised) was carefully kept at 9 grains per gallon (153 ppm), with a molar ratio of  $\text{Ca}^{2+}/\text{Mg}^{2+}$  equal to 3:1. The concentration of copper ( $\text{Cu}^{2+}$ ) ions was kept at about 1 ppm (+/- 10%), the exact concentrations being measured by a standard analytic method. The rinsing water flow rate was 6 liters per minute. The same shampooing and rinsing process was repeated another time. This treatment cycle was repeated for five cycles total, and was the same as the one described as the "5-Cycle Oxidative Hair Treatment Protocol With 2 Intermediate Washes" in the present application.

Measurement of colour and lightening was performed on a Hunter Labscan Spectrophotometer. Details on this equipment are given in Appendix 2.

Fibre damage was measured using an FT-IR method and analyzing the  $1040\text{ cm}^{-1}$  peak, which correlates with the formation of cysteic acid. Details on the equipment used and the test protocol are given in Appendix 3.

## RESULTS

Table 1 below shows the lightening values for the hair after 5 cycles of treatment expressed as dL (i.e., the increase in lightening after 5 cycles), and the cysteic acid as measured by FT-IR. Also included is a damage/lightening ratio, as it was noted that all of the different systems gave different amounts of lightening.

Table 1. - Lightening & FT-IR Damage Values

	Product	Lightening (dL)	FT-IR Cysteic Acid Damage	Damage / dL Ratio
1	pH 8 / Peracetic acid + hydrogen peroxide + 1.9% EDDS	8.3	111	13.7
2	pH 8 / Peracetic acid + hydrogen peroxide + 0.1% EDTA	11.7	186	15.9

3	pH 10 / Peracetic acid + hydrogen peroxide + 0.1% EDTA	19.2	173	9.01
4	pH 10 / hydrogen peroxide + 2.0% EDTA	22.4	165	7.37
5	pH 10 / hydrogen peroxide + 1.9% EDDS	22.2	110	4.95

### CONCLUSION

It is the Examiner's opinion that U.S. Patent No. 6,004,355 to Dias et al. ("Dias") teaches hair dyeing compositions comprising the same ingredients in the same amounts as the compositions of the present application, and, therefore, the hair dyeing compositions of Dias would inherently possess the same physical properties as the compositions of the present application, such as a damage benefit as claimed. Based upon the results of the experiments described above, it is my conclusion that hair dyeing compositions of Dias, which are required to contain a peroxyacid precursor oxidizing aid, do not possess the same damage benefit as compositions of the present application. As can be seen in the results above, the hair dyeing compositions at pH 8, based on the exemplified compositions of Dias, had significantly less lightening than the compositions only containing hydrogen peroxide (when lightening is measured at equal oxidant levels). In addition, the hair damage was significantly higher when measured per unit of lightening achieved for the peracetic acid / hydrogen peroxide systems at both pH 8 and pH 10, as compared to the compositions containing only hydrogen peroxide and at pH 10. Most notably, Product 3, which was based on the exemplified compositions of Dias and was at pH 10, provided a damage benefit of 173 cysteic acid units. This damage benefit level is greater than the claimed damage benefit of less than about 160 cysteic acid units. Therefore, it cannot be assumed that the compositions of Dias inherently possess the same damage benefit properties as the compositions of the present application.

This declaration is made with the knowledge that all statements made herein of my own knowledge are true, and that all statements made on information and belief are believed true, and further that willful false statements and the like are punishable by fine or imprisonment, or both under 18 USC §1001 and may jeopardize the validity of the application or any patent issuing thereon.

29th March 2005

Date

Jennifer Mary Marsh

Jennifer Mary Marsh

Declarant